

09/25/474

PATENT SPECIFICATION

(11) 1369 269

1369 269

(21) Application No. 4425/72 (22) Filed 31 Jan. 1972
 (31) Convention Application No. 111556 (32) Filed 1 Feb. 1971 in
 (33) United States of America (US)
 (44) Complete Specification published 2 Oct. 1974
 (51) International Classification C11D 11/04//B01F 7/04
 (52) Index at acceptance

B1X 17
 B1C 10 14 18G5A 18G5B 1 25 4 X26
 C5D 6A5B 6A5D1 6A5D2 6A9 6B12B1 6B12G2A 6B4
 6B9 6C6 6D



(54) METHOD FOR NEUTRALIZATION OF DETERGENT ACID

(71) We, COLGATE-PALMOLIVE COMPANY, a Corporation organised and existing under the Laws of the State of Delaware, United States of America, of 300 Park Avenue, New York, New York 10022, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the neutralization of synthetic organic anionic detergent acids.

In the manufacture of synthetic organic detergents of the anionic type, a detergent acid is often produced initially and is then neutralized, usually in aqueous solution and with an aqueous neutralizing agent. To utilize the detergents that are so produced in solid, particulate or powdered products, it is generally necessary to dry them or take up the aqueous solvent as water of hydration for a hydratable material, such as a builder or filler salt. Prior attempts to avoid having to undertake such additional processing steps by neutralizing directly with solid, particulate or powdered neutralizing agents have encountered various difficulties. For example, local discoloration of the organic detergent may occur. Also, the product, although appearing to be dry and uniform, may contain soft spots within the neutralized detergent particles. In addition, the materials may adhere both to processing equipment walls and to other particles, and become plasticized, causing undesired deposits, lumping, high power consumption in mixing and grinding, etc.

The present invention provides an efficient, rapid, economical and commercially practicable method for dry-neutralizing such acids, which produces a light-coloured product of excellent quality which is free flowing and capable of being pulverized without blocking equipment openings or creating power overloads on the equipment.

In accordance with the present invention, a method of neutralizing a synthetic organic anionic detergent acid comprises rapidly mov-

ing neutralizing agent in powder form in a reaction zone while the acid is being added thereto, blowing a gas through the moving mixture of neutralizing agent, acid and neutralized detergent, moving the mixture past cutting means, continuing the mixing, cutting and gas blowing of particles and any aggregates of neutralizing agent, acid and neutralized detergent during, or during and after the addition of the acid, and removing from the reaction zone the gas and at least some of the water vapour resulting from the neutralization reaction.

In preferred embodiments of the invention, the detergent acid is a straight chain alkyl benzene sulphonate acid, the neutralizing agent is sodium carbonate, the gas employed is air and the acid is added to the powdered neutralizing agent, which is in continuous rapid motion, over a short period of time, usually about 5 minutes or less. The product produced should usually contain from 25 to 35% of the synthetic organic detergent salt for best results.

It is also preferred that the sodium carbonate neutralizing agent has mixed with it, before addition of the detergent acid, a building or filler salt.

The temperature to which the reaction mix is raised during the neutralization may be in the range 50 to 110°C, or even higher, although the temperature will usually not be higher than 100°C.

Blowing is preferably continued after completion of the addition of the detergent acid.

The product may be employed as a detergent composition or as a detergent component of other materials, such as foaming scouring cleansers.

The accompanying drawings illustrate, by way of example, one form of apparatus by which the method may be performed. In the drawings:

Figure 1 is a front perspective view of the apparatus;

Figure 2 is a rear perspective view of the apparatus;

50

55

60

65

70

75

80

85

90

Figure 3 is a partial sectional view along plane 3—3 of Figure 1;

Figure 4 is a partially cutaway side elevation showing the flows of reagents and product; and

Figure 5 is a schematic view and flow diagram showing the neutralizing apparatus and auxiliary equipment used to feed it and to process the neutralized product.

A high shear mixer 11 of the horizontal cylindrical type, the basic part of which is commercially available from Littleford-Lodige Division of Littleford Bros. Inc., Cincinnati, Ohio, U.S.A., and which is known, in unmodified condition, as a Lodige mixer, comprises a horizontal cylindrical drum 13, a pair of ploughs 15 and 17 driven by a main motor 19, a pair of end scrapers 21 and 23 also driven by the main motor 19, a pair of choppers 25 and 27 driven by individual chopper motors 29 and 31, access doors 33 and 35, a charging port 37, a contoured outlet door 39 at the bottom of the cylinder and a vent port 41. As illustrated, the cylindrical drum is covered with an insulated jacket 43 through which may be passed heating or cooling fluid to regulate the temperature of materials inside the mixer. For the present method, it has been found that such temperature control is not necessary, apparently partly due to self-regulating effects caused by the evaporation of moisture from the mixture. Indeed, a better product often results when cooling water is not used, although one would normally expect to have to employ cooling water to prevent excessive heating of the exothermally reacting mixture.

The neutralizing material, e.g. sodium carbonate, is employed in excess and is maintained in a rapidly moving mass, intimately mixed with air, the mass being hurled rapidly against the droplets or stream of detergent acid entering the mixer. Thus, the main driving motor 19 causes the ploughs and scrapers, 15, 17, 21 and 23 to rotate at a speed in the range 100—150 r.p.m., e.g. at 130 r.p.m., which causes the neutralizing agent to be mixed thoroughly with detergent acid and with partially neutralized detergent material and lifted upwardly into the "air space" about twice every second. Compressed air, at a pressure in the range 3 to 5 p.s.i.g., is employed to keep particles out of and to flush the various bearings of the mixer, by being forced through clearances therein, as at 45, 47, 49, passing from a source, not shown, through piping 51, 53, and 55, respectively. Such compressed air ultimately passes into the mixer 11 but usually is insufficient in quantity to maintain the solid neutralizing salt in a fluidized or aerated state as a swirling mass of individual particles buoyed up by the air. A separate air line 57, which may be equipped with a diffuser 59 having openings 61 therein may be used to distribute air through the

materials being mixed. Such materials are maintained in circulation by the combination action of the ploughs, the choppers, air and, to some extent, the scrapers. The pressure in the mixer is maintained at about atmospheric by means of an exit vent 63 having at the top thereof a permeable bag 65 or a plurality, e.g. two, of bags through which air will pass but solid particles will not.

The solid neutralizing agent, in powder form, may be added to the mixer through the access door or in any other suitable way, but it is normally desirable that this be done automatically through a feeding device and a closed chute or duct 67, to avoid excessive dusting in the vicinity of the mixer. Synthetic organic detergent acid is admitted to the mixer through lines 69 and 71 and orifices 73 and 75, respectively. The points of addition of the acid, which enters the mixer in fine stream or droplet form, are near the path of the neutralizing powder where the powder is in vigorous motion due to the actions of the ploughs 15 and 17 and the choppers 25 and 27. Thus, with the choppers, ploughs and scrapers in operation and with air being blown into the mixer, a fine dispersion of the neutralizing powder in air is brought into contact with the detergent acid at a very high speed and neutralizes the acid. The speeds of the chopper blades, of which there are usually from two to six per chopper, are in the range 1,000 to 5,000 r.p.m., preferably about 3,600 r.p.m. Partially neutralized detergent acid is thus intimately mixed with the neutralizing agent and is thrown into the paths of the chopper blades again, so that there are repeated dispersions of such material and little opportunity for there to be an excess of acid in contact with partially neutralized detergent for any appreciable period of time. The air continually removes moisture and heat from the neutralized detergent. Furthermore, some of the moisture present, which could otherwise tend to make the detergent salt tacky, is physically absorbed or serves to hydrate excess carbonate or other neutralizing agent or builder salt. It may also be scavenged by calcium oxide powder, forming calcium hydroxide. Thus, the product is in finely divided form and is free flowing. It is not discoloured or tacky due to local overconcentrations of detergent acid or contents of only partially neutralized detergent salt. It is suitable for blending with other detergent materials and it is especially useful as the organic detergent constituent of abrasive cleansers, such as scouring powders.

In some cases it will be desirable to pulverize the neutralized detergent further so that its particle size will approximate that of a product into which it may be incorporated, e.g. very finely powdered abrasive particles of silica flour. Because it is completely neutralized and free flowing, the detergent salt

may be easily size reduced in such equipment as a micropulverizer without blocking the screen thereof or requiring excessive power consumption to effect pulverization.

5 Figure 4 illustrates the mixing action which takes place in the modified Lodige mixer. As shown, the "bed" of neutralizing agent, mixed with builder salt, is circulated in the mixer by the movements of the ploughs, scrapers and chopper blades, plus the air being injected into it. The liquid synthetic organic detergent acid, added in streams near the chopper blades, is immediately broken into contact very fine particles and thrown into contact 10 with the particles of moving solid neutralizing agent. The paths of the materials are everchanging but that illustrated is representative of a typical pattern.

15 Figure 5 is a schematic flow diagram of an installation for producing a scouring cleanser, starting with detergent acid, neutralizing agent, inorganic builder, silica powder and adjuvants. Acid mix, produced by the sulphonation of C_{10} — C_{20} linear alkyl benzene, is stored in a tank 77, from the bottom of which an inorganic (sulphuric acid) layer may be withdrawn and discarded. Such settling operation is not necessary with a detergent acid made by sulphur trioxide sulphonation.

20 The acid mix is led to a feed tank 79 calibrated to store the exact batch weight desired, and from there is discharged when when needed through a pump 81 into the modified Lodige mixer 11. At the time of addition of the acid, the mixer will be in operation, with the choppers, ploughs and scrapers maintaining the neutralizing agent and builder, if any, in a state of continual movement and dispersion. If desired, addition of air may be effected only after the neutralizing powdered material is sufficiently wetted or increased in density by the acid mix so as to be less apt to rise and plug the vent bag 65. The solids are first added to 25 the mixer 11 by being weighed and dumped into Redler conveyor 83 at a dump station 85, from which they are transported through a chute 87 to the mixer 11. After completion of neutralization of the detergent acid, the product may be dropped through a hopper 89 onto a vibratory feeder 91, through a micropulverizer 92, a chute 93 and a 3 way diverter gate 95 into either of two mixers 97 and 99. Alternatively, the finely divided 30 neutralized detergent may be removed through a line 101. The mixers may be ribbon mixers, and serve to disperse the detergent solids in a major proportion of silica powder and produce a foaming detergent scouring cleanser.

35 Various adjuvants may be added at any suitable point of this apparatus. For example, there may be added together with the silica powder a bleaching agent and a perfume, to produce a bleaching and foaming detergent 40 scouring cleanser.

The dry method of neutralizing detergent acid as described above is superior to various wet neutralization methods wherein the detergent slurry produced must thereafter be dried. It also minimizes contact of the alkyl aryl sulphonate salt with very hot surfaces, as in a spray tower, where it can char or actually ignite. By the present method, effective neutralization is obtained without product degradation or discoloration.

45 Although the method is applicable to neutralizations of various detergent acids, it is most useful in effecting such reactions with alkyl benzene sulphonics acids wherein the alkyl group is 10 to 20 carbon atoms, preferably from 10 to 18 carbon atoms and most preferably from 12 to 15 carbon atoms. Although branched chain alkyl benzene sulphonics acids may be employed, it is preferred to employ the straight chain compounds, e.g. linear dodecyl benzene sulphonics acid. The linear alkyl benzene sulphonates, especially those with the alkyl group terminally attached to the benzene or attached at the 2-carbon are found to produce a dry and free flowing product.

50 The alkyl aryl sulphonics acids are preferably converted to the water-soluble, normally solid salt form, and of the salt, the alkali metal salts, especially the sodium salts, are preferred.

55 The neutralizing agent employed may be any suitable alkaline compound which does not result in an undesirable by-product during the neutralization of the detergent acid. Thus, for example, sodium hydroxide sodium phosphate, borax, pentasodium tripolyphosphate, tetrasodium pyrophosphate, disodium phosphate, potassium phosphate, sodium bicarbonate, sodium carbonate, sodium silicate, calcium oxide, calcium hydroxide and mixtures thereof may be used.

60 Among the builder and filler salts that may be employed with the neutralizing agent, or which may be used as both builders and neutralizing agents, are included the previously mentioned neutralizing compounds except for the hydroxides. The builders improve the surface active and deterutive properties of the detergent salts and are especially useful in the making of heavy duty synthetic detergent compositions. Even when they do not perform a primary neutralizing action in the present method, they are often useful in the neutralizing reaction to disperse the reactants, to absorb moisture and to make the product more freely flowing. Calcium oxide is very effective as such a builder. Instead of or in addition to builders, other useful materials and adjuvants, including filler salts may be present. Thus, sodium sulphate or sodium chloride may be used, and in some cases it may be desirable, where the end use for the neutralized detergent is in an abrasive composition, such as a scouring cleanser, to have silica or other abra-

sive mixed in with the neutralizing agent and/or builder in the mixer.

Various adjuvants may be included with the neutralizing agent in the mixer, providing they are non-interfering with the neutralization reaction and are not adversely affected by contact with the reagents or products. Thus, stain-removing chemicals, oxidizing agents, reducing agents, perfumes, colourants, bactericides, fungicides, foaming agents, solvents, bleaches, brighteners, enzymes, bulking materials and pH controllers may be used.

The alkyl aryl sulphonates and the builders described above make presently preferred products, but other synthetic detergent acids may be neutralized by the present method and the resulting salts may be utilized in place of or in partial substitution for the alkyl aryl sulphonates. Thus, for example, olefin sulphonic acids, olefin disulphonic acids, higher alkyl sulphuric acids and polycarboxylated higher alkyl sulphuric acids may be neutralized by the present method and the salts made may be incorporated in the final products.

Excepting the adjuvants, which will usually comprise a minor proportion, generally less than 5% each with a total adjuvant content of less than 20%, the proportions of reagents employed will usually be those set forth below.

Usually, but not always, the neutralizing agent, preferably sodium carbonate, will be used in excess and the final product will contain an appreciable amount thereof. The synthetic organic detergent salt made will generally be a minor proportion of the finished composition discharged from the mixer. The inorganic builder salt present may be either a major or minor proportion of the product. In preferred compositions, which comprise the sodium salt of linear alkyl benzene sulphonic acid, sodium carbonate (and/or sodium bicarbonate by-product) and builder salt, the organic detergent component, including any other organic detergent neutralized with the linear alkyl benzene sulphonic acid, will be from 25 to 45% of the dried composition from the mixer-neutralizer. Preferably, it will comprise only sodium alkyl benzene sulphonate wherein the alkyl group is straight chain and of 12 to 15 carbon atoms and that detergent will be present as 25 to 35% of the product. Of course, lower proportions of detergent may be used in the method of the invention, as low as 20% and even 5%, but then the advantages of the present method, compared to other dry neutralization methods, will not be as significant, largely because the difficulties to be overcome will not be as severe. The sodium carbonate content of the final product, including an equivalent weight of any carbonate that had been converted to bicarbonate (usually, there is little bicarbonate in the final product) will be from 0 to 75% of the product, preferably from 10 to 40% and most preferably from 20 to 35% thereof. The

builder (builder salt such as trisodium phosphate, or organic builder compound) should be from 0 to 70%, preferably from 25 to 45% and most preferably from 30 to 40% of the dried product. The above percentages are on the basis of an "as is" product, including any moisture in the solid product, which may result from water present in the solution of detergent acid and from the neutralization reaction. Generally, it has been found that the proportion of moisture present in the product in free non-hydrate form should be less than 5% and in the preferred compositions it is less than 2%. Of course, if some of the moisture is held by a strong and non-tacky hydrate, e.g. calcium hydroxide (from the oxide), a greater percentage, e.g. up to 10%, can be tolerated in the product. Most preferably, especially in products not containing hydratable salts or water absorbents, moisture contents are as low as feasible, even under 1% or under 0.5%, because the presence of moisture, even in very small amounts, contributes to plasticity, tackiness, adhesion and lumping.

It has been found that the detergent acid, in liquid form, containing a small but sufficient amount of moisture to promote reaction with the neutralizing agent, should be added to the mass of rapidly moving solid neutralizing agent. If the reverse operation is attempted, wherein the acid is introduced first into the mixer and has neutralizing agent and builder salt added to it, the liquid droplets tend to coalesce and form films or pools in the mixer, leading to uneven neutralization.

The volume of the mixer should not be completely filled with reagent solids or liquids. Space should be left for a gas or gases to circulate through the reagents, whereby excessive heat build-up is avoided, the exothermic reaction is tempered and moisture is evaporated and removed with the outgoing gas. Also, such free volume allows a place for the fine neutralizing salt and builder powders to move when mixing is begun, thereby preventing the powders from being carried over into the venting bag where, if the bag were moist, it could become plugged with powder, necessitating shutting down for cleaning. The "free volume" in the mixer will normally be from 20 to 80%, preferably from 50 to 75% and most preferably from 60 to 70% thereof.

The gas, which together with the mixing and/or chopping action of the modified Lodge mixer creates the desired turbulence in the mixer, is preferably air but can be any other dry gas which is inert with respect to the materials in the mixer. Thus, nitrogen, oxygen, hydrogen, argon and low boiling halocarbons, e.g. dichlorodifluoromethane, may be used. Generally, the gas will be used at temperatures in the range from 60 to 95°F, although in some cases colder or warmer gases may be charged. Dried or warmed gas can be

employed so as to increase its capacity to remove moisture. Air flow rates are usually from 5 to 50, preferably 10 to 30, cubic feet per minute. The volume of gas charged to the mixer will generally be such as to change the gas content of the mixer at least once every two minutes and preferably such a change will be made within from 10 to 60 seconds. In addition to the main inlet for compressed gas, which is usually a pipe of from 1/8 to 2 inches inside diameter, depending on the mixer volume, through which air at a pressure of from about 5 to about 200 p.s.i.g., passes, other sources of gas to the mixer include that used for flushing the mixer, scraper and chopper bearings. The flow rate of air through the mixer is regulated also by the resistance of the venting bag or other dust collecting equipment, which prevents solid materials from being discharged with the air. The pressure in the mixer will be close to atmospheric, generally being up to 5 p.s.i.g., preferably from 0.1 to 1 p.s.i.g.

The speeds and forms of the chopper blades, ploughs and scrapers are such that there is no bed of unchanging reagents or product created at the bottom of the mixer. Rather, the mass is in constant motion and the particles move at speeds of about 200 to about 5,000 feet per minute.

The temperature in the mixer, initially at about room temperature, rises due to the exothermic reaction. Because heated detergent acid and partially neutralized detergent have generally been considered to be tacky products which should be avoided at all costs, during experimental work cooling water was used in the mixer jacket to hold the reaction temperature down. However, it was found that with the present method excessive tackiness does not result, and it is even advantageous for the temperature to be allowed to rise because the moisture content of the product is thereby diminished due to evaporation at elevated temperatures. Thus, it is unnecessary to use cooling water. In fact, if cooling water is employed, water vapour may condense on the internal walls of the mixer. This can form a cake or scale with the phosphate and carbonate, which adheres to the mixer walls and interferes with the plough and scraper movement. This leads to greater power consumption, less efficient mixing and a poorer final product.

The temperature of the acid mix, neutralizing agent and builder salt charged will usually be in the range of from 10 to 70°C, preferably from 40 to 70°C for the acid. The higher temperatures are especially preferable for the acid and assist in effecting fast reaction and in removing water. During the neutralization reaction, which takes from 1 to 16 minutes, preferably from 2 to 10 minutes and most preferably from 4 to 8 minutes, including addition of acid mix, which gener-

ally requires from 1 to 2 minutes, and a subsequent additional mixing period, the temperature will normally rise by about 40 to 80°C, usually to about 50 to about 110°C. After addition of the acid mix, mixing is continued for another 30 seconds to 5 minutes, preferably for at least 2 minutes, after which the neutralized product is removed from the mixer and is processed further. At such time it is free flowing, although usually at a high temperature, e.g. 75—80°C, and it has a comparatively low moisture content, generally from 0.1 to 3%, preferably from 0.1 to 2%. This additional mixing, while assisting completion of the reaction, also allows the temperature of the reaction mix to fall by a few degrees, generally from 3 to 10°C, due to the cooling action of the air passing through the mixer. The fall in temperature is also indicative of the end of the exothermic reaction.

After completion of the mixing, the product is dropped to a size reducing machine, such as a micropulverizer, which reduces it to the desired particle size range. As dropped from the neutralizer-mixer, the product may include as much as 50% by weight of particles on a U.S. Sieve No. 20, although usually the particle sizes will be smaller. At the comparatively high temperature at which the particles are dropped to the pulverizer, they are stable and non-tacky and can be reduced to a very fine powder, substantially all of which passes through a 200 mesh screen (U.S. Sieve). The product is suitable for direct mixing with very finely divided abrasive particles and polishing agents, e.g. talc or silica. Thus, no additional grinding of the abrasive-detergent mixture is needed to avoid settling out of some of the components of such a product.

Among the many advantages of the present method, in addition to those mentioned above, are the surprising stability of the products with respect to bleaching agents, such as trichloroisocyanuric acid, and to other materials which are unstable in the presence of moisture and organic constituents, plus its good storage characteristics, it being substantially non-caking over long periods of storage at comparatively high temperatures and humidities. The method is adaptable for a wide variety of production rates, unlike the conventional spray towers which normally have to have high volume utilization to be economic. Little floor space is required for the apparatus and essentially no storage space is needed because the neutralized detergent composition can be incorporated into scouring cleansers or other products immediately after manufacture. Production of burned or charred material, such as often results from spray drying operations, is eliminated by the present method. Also, there is no need for the combustion of large amounts of fuel. The method is also a speedy

one and allows rapid neutralization of the detergent acid.

The following Examples further illustrate the invention. Percentages in the Examples and elsewhere in the specification are by weight.

EXAMPLE 1

A neutralized linear dodecyl benzene sulphonate, sodium salt, is prepared by the dry neutralization of the corresponding water-washer detergent acid with sodium carbonate in the presence of trisodium phosphate in a high shear mixer of the Lodige mixer type, model FKM 600D, modified to include lines 10 for adding detergent acid and for forcing compressed air into the mixer, and having a venting sock (or a pair of such socks) at the top. The apparatus is made of stainless steel and includes a contoured bottom outlet door, 15 a charging port, access doors and a vent port so that the smooth-walled cylindrical shape of the mixer body is maintained. It also has two scrapers and five ploughs driven by a 15 horsepower motor at 130 r.p.m., and two four-blade choppers, which may be of six to eight inches in diameter and are each driven by separate 5 horsepower motors at 3,600 r.p.m. Air purge seals, with air supplied at 3 20 to 5 p.s.i.g., are provided to flush the main bearings and chopper bearings. The mixer has approximately 22 cubic feed total capacity and the active working capacity is about 13 cubic feet. A water jacket is provided but cooling water is not employed in the present example.

Batch weights of light soda ash, 76.5 lbs. at 99.2% purity, trisodium phosphate, 112.3 lbs., and preservative 0.1 lb., are weighed out and conveyed to the mixer by a Redler conveyor. This chain conveyor is four inches in width and is equipped with a vented dumping station. After the high shear mixer is charged with the particulate solids, the ploughs, 40 choppers and scrapers are started and acid addition is begun. This is accompanied by only shaft seal air addition. After one fourth of the acid has been added, air flow is begun and the pressure of the air in the inlet pipe is increased to about 30 p.s.i.g. to provide a 45 pressure in the mixer of about 2 p.s.i.g. Thus, the air pressure is held low so that air flow is about 10 c.f.m. The acid addition amounts to 76.5 lbs. of water-washed linear dodecyl benzene sulphonate acid mix which is added over a four minute period, with the air pressure being increased to give a flow of 20 c.f.m. after one minute. The water-washed acid mix comprises approximately 86.8% of active 50 detergent acid, 7.8% of sulphuric acid, 1.3% of free oil and 4.1% of water. After completion of the addition of the acid mix, mixing is allowed to continue for two minutes, after which the mixer is halted and the product is dropped out, to be size reduced and incorporated

into a scouring cleanser as the built organic detergent component thereof. During the neutralizing process the temperature rises to about 85°C and during the mixing after addition of the acid mix the product temperature drops by about 5°C to about 80°C.

The product removed is of good colour and shows no signs of deterioration of the detergent. It is substantially free flowing and capable of being size reduced without blocking the pulverizing equipment screens. Although not as finely divided as the initial materials charged, the product is in particulate form with the following approximate particle size distribution:

Mesh Size Standard (U.S. Sieve)	Sieve Analysis		Percent	80
	on	Percent		
on 8		0.3		
on 12		1.6		
on 20		11.8		85
on 30		12.9		
on 40		10.2		
on 60		15.0		
through 60		48.2		

Thus, over 60% of the product passes a No. 40 sieve and over 50% will not pass through a No. 60 sieve. In some cases the product is even finer than that indicated above. The moisture in the built detergent particles is from 1.5 to 1.8%, by oven drying determinations. Thus, the evaporation of significant amounts of moisture in the mixer are apparent.

After dropping of the batch is it conveyed to a size reducing machine, preferably a hammer mill equipped with herringbone screens, and reduced to the following particle size distribution:

Mesh Size (U.S. Standard Sieve)	Percent	105
on 20	nil	
on 40	0.3	
on 60	3.5	
on 80	8.6	
on 100	7.6	
on 125	32.0	110
on 325	27.9	
through 325	20.1	

Analysis shows the product to comprise 26.9% of linear dodecyl benzene sulphonate, sodium salt, 18.2% phosphate, as P_2O_5 , and 1.6% moisture. As such, it is an excellent source of built synthetic organic detergent for the manufacture of scouring cleansers, which comprise major proportions of abrasives or polishing agents, such as silica powder. In a large ribbon mixer, 2,560 lbs. of fine silica powder are mixed with about 10 lbs. of perfume, 258 lbs. of the neutralized built detergent salt made according to the described

method and 14 lbs. of trichloroisocyanuric acid. The perfume is blended in with the silica powder after about half of the silica powder has been added to the mixer. After the perfume is dispersed sufficiently, the neutralized detergent and trichloroisocyanuric acid are added and blended in sequentially. There is no need for any further size reduction and the product is dropped from the ribbon mixer after a few minutes mixing, usually from 5 to 10 minutes, and may be filled into containers. It will normally contain about 2.5% of active detergent salt, 1.6% of total phosphate, as P_2O_5 , and 0.4% of available chlorine, in the bleaching agent. Stability tests run on this material show that it does not cake and continues to flow freely after storage at high temperatures and at high humidities. Also, the chlorine content remains high during storage. The product seems to flow more freely than one made with a spray dried detergent and its volume is slightly greater. Costs of manufacture are substantially lower, both with respect to capital expenditures and labour charges. Approximately 50 batches can be made per 8 hour shift, by two operators. Production is efficient and trouble-free, even on warm, humid days.

EXAMPLE 2

A neutralized linear dodecyl benzene sulphonate, sodium salt, is prepared by dry neutralization of the corresponding sulphonated and water washed detergent alkylate with the stoichiometric amount of sodium carbonate in the presence of a builder proportion of pentasodium tripolyphosphate, using the equipment described in the foregoing Example. Batch weights of light soda ash, 17.6 lbs. of 99.2% purity, and 171 lbs. of pentasodium tripolyphosphate are weighed out and carried to the mixer by the conveyor. When the powders are charged the ploughs, scrapers and choppers are started and air is blown into the mixer at a low pressure to produce a flow rate of about 10 c.f.m. Then, with the powders being agitated, 76 lbs. of the detergent acid are charged to the mixer over a 1 minute period. Neutralization of the acid occurs shortly after contact with the mixed alkaline salts.

After passing through the mentioned size reducing equipment of Example 1, the finished product is of good colour, is free blowing and closely resembles the product of Example 1 in appearance, particle size distribution and handling properties. It is 27.5% linear dodecyl benzene sulphonate, sodium salt; 36.6% phosphate, as P_2O_5 ; 1.7% moisture; and contains a trace of unreacted sodium carbonate.

The composition is useful as a source of active ingredient in products which usually contain substantial percentages of pentasodium tripolyphosphate, such as hard surface cleaners

and dry bleaches in which the active bleach is potassium dichlorocyanurate.

65

EXAMPLE 3

A neutralized branched chain dodecyl benzene sulphonate, sodium salt, is prepared by dry neutralization of the corresponding dodecyl benzene sulphonate resulting from the sulphur trioxide sulphonation of tetrapropylene benzene. The neutralizing agent is sodium carbonate powder. No other neutralizing agent or builder is present. Neutralization is effected in the equipment previously described, in Examples 1 and 2.

The batch weight of sodium carbonate, 188 lbs., of 99.2% pure and dry material, is weighed out and charged to the modified Lodge mixer described. With the ploughs, scrapers and choppers in operation, the detergent acid is fed to the mixer onto the swirling sodium carbonate particles. Acid addition takes about 1 minute and after it is about half complete the air valve is opened to allow about 10 c.f.m. of air to blow through the mixer and the moving mass of reactants. Upon completion of the addition of the acid the air ratio is increased to about 20 c.f.m. Mixing continues at the increased air flow rate for about another 6 minutes. The temperature of the mixer increases to about 85°C during the reaction and then drops to about 80°C thereafter.

The product is pulverized to substantially the same particle size distribution as that of Example 1 and is suitable for use in many types of cleansers and cleaners. Its composition is 27.5% branched chain higher alkyl benzene sulphonate, sodium salt; 1% moisture; 0.5% sodium sulphate; and the rest sodium carbonate. The detergent powder made is useful in finished products in which phosphates are not desirable ingredients or wherein the phosphates should not be present during size reduction, neutralization or other operations, although they may be added at final compounding.

WHAT WE CLAIM IS:—

1. A method of neutralizing a synthetic organic anionic detergent acid, comprising rapidly moving neutralizing agent in powder form in a reaction zone while the acid is being added thereto, blowing a gas through the moving mixture of neutralizing agent, acid and neutralized detergent, moving the mixture past cutting means, continuing the mixing, cutting and gas blowing of particles and any aggregates of neutralizing agent, acid and neutralized detergent during, or during and after the addition of the acid, and removing from the reaction zone the gas and at least some of the water vapour resulting from the neutralization reaction.

2. A method according to claim 1 wherein the gas is forced through the mixture in an

65

70

75

80

85

90

95

100

105

110

115

120

125

amount sufficient to change the gas content of the mixer at least once every two minutes.

3. A method according to claim 1 or claim 2 wherein the neutralizing agent is maintained in continuous motion during the neutralization reaction by the actions of mixing means, cutting means and gas movement through it.

4. A method according to any of the preceding claims wherein the mixing, cutting and gas blowing are continued for at least 30 seconds after completion of the addition of the acid.

5. A method according to claim 4 wherein the mixing, cutting and gas blowing are continued for at least 2 minutes after completion of the addition of the acid.

6. A method according to any of the preceding claims wherein the temperatures of the neutralizing agent and the acid are both initially in the range of 10 to 70°C, and the temperature rises during neutralization to within the range 50 to 110°C.

7. A method according to any of the preceding claims wherein the neutralizing agent is present in excess of the stoichiometric amount required for neutralization of the acid and there is unreacted neutralizing agent present in the product.

8. A method according to any of the preceding claims wherein the acid comprises an alkyl benzene sulphonic acid and the neutralizing agent comprises sodium carbonate.

9. A method according to claim 8 wherein the acid is a linear alkyl benzene sulphonic acid in which the alkyl group is of 12 to 15 carbon atoms.

10. A method according to any of the preceding claims wherein the neutralizing agent has mixed with it a builder salt in powder form.

11. A method according to claim 10 in which the builder salt comprises trisodium phosphate.

12. A method according to claims 9 and 11 wherein the proportions of materials charged are such as to result in a product comprising from 25 to 35% of sodium alkyl benzene sulphonate wherein the alkyl group is linear and of 12 to 15 carbon atoms, 25 to 45% trisodium phosphate and 10 to 40% sodium carbonate, by weight.

13. A method according to any of the preceding claims wherein the gas is air.

14. A method according to any of the preceding claims wherein the product is removed from the mixing-neutralizing zone and is pulverized.

15. A method according to claim 14 wherein the pulverized product is mixed with a major proportion of silica powder, to produce a foaming detergent scouring cleanser.

16. A method according to claim 14 wherein the pulverized product is mixed with other substances comprising bleaching agent, perfume and a major proportion of finely divided silica powder, to produce a bleaching and foaming detergent scouring cleanser.

17. A method of neutralizing a synthetic organic anionic detergent acid, substantially as described with reference to the accompanying drawings.

18. A method of neutralizing a synthetic organic anionic detergent acid, substantially as described in any of the Examples.

19. A synthetic organic anionic detergent salt which has been made by neutralization of a synthetic organic anionic detergent acid by a method according to any of claims 1 to 14, 17 or 18.

20. A method of making a detergent scouring cleanser substantially as described with reference to Figure 5 of the accompanying drawings.

21. A method of making a detergent scouring cleanser substantially as described in Example 1.

22. A detergent scouring cleanser which has been made by a method according to any of claims 15, 16, 20 or 21.

KILBURN & STRODE,
Chartered Patent Agents,
Agents for the Applicants.

Printed for Her Majesty's Stationery Office, by the Courier Press, Leamington Spa, 1974.
Published by The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from
which copies may be obtained.

1369269 COMPLETE SPECIFICATION

2 SHEETS This drawing is a reproduction of
the Original on a reduced scale

Sheet 1



